Figure 4. Plots of $^{236}U/^{238}U$ ratios versus $^{238}U/^{235}U$ ratios. Upper figure shows data for all samples, lower figure shows a greatly expanded scale to facilitate examination of the bulk of the data. Two-sigma errors are shown. Samples discussed in the text are labeled. Mixing lines between U with natural isotopic composition and the samples that vary substantially from natural are shown on both figures. Note that 8802D filter in the lower diagram plots along the mixing line between natural U and 8802D water, suggesting that the measured $^{236}U/^{238}U$ ratios are accurate. The $^{236}U/^{238}U$ for the rest of the samples in the lower figure are considered below the detection limit. See text for further discussion.

Six additional samples plot slightly off of the natural U line (Fig. 2). Samples W23L08 (water and filter) and SDA10 (water) are from the vadose zone, 8802D filter is from a perched water zone and sample USGS 120 (water and filter) is an aquifer sample.

In order to evaluate if the $^{238}U/^{235}U$ ratios of these six samples do actually deviate from that of natural U, the overall accuracy of this data set must be assessed. To do so, we make the assumption that the remainder of the data represents water with natural U. We exclude samples PA02 Filter, SDA8 Filter, SDA10 filter and W25 filter because of the high uncertainties of these analyses. The mean $^{238}U/^{235}U$ of the remaining 36 samples is 137.83 with a standard deviation of 0.23. This mean and ± one standard deviation are plotted in Figure 2. This value is taken as a conservative assessment of our accuracy of $^{238}U/^{235}U$ ratio measurements for this study. Given this, then the $^{238}U/^{235}U$ ratios from samples W23L08 water, USGS 120 water and filter, and SDA10 water overlap the natural U line. This evaluation is supported by results of a statistical test using Chauvenet's Criterion, which includes these four samples with the bulk of the data set. In addition, the $^{236}U/^{238}U$ and $^{234}U/^{238}U$ ratios, as well as total U concentrations of these samples do not indicate the presence of anthropogenic U. Thus, these data are interpreted to indicate that the measured U isotopic ratios for samples W23L08 water, SDA10 water, and USGS 120 water and filter are natural.

The statistical test using Chauvenet's Criterion identifies two samples, 8802D filter and W23L08 filter, as different from the rest of the data set. These two samples are considered possible candidates for containing a component of anthropogenic U. These two samples plot more than 3-sigma above the natural U line (Fig. 2), with $^{235}U/^{238}U$ ratios of 138.60 ± 0.15% and 139.33 ± 0.20% respectively. The $^{234}U/^{238}U$ as well as total U concentrations of these samples are well

within the range of values determined for this study. The $^{236}U/^{238}U$ ratios for these samples are low and well within the range of values considered as background for this study. The $^{236}U/^{238}U$ ratio for sample 8802D filter; however, plots on a mixing line between natural U and its water counterpart (Fig. 4) suggesting that the $^{236}U/^{238}U$ ratio measured for this sample may be accurate. Taken together, the $^{238}U/^{235}U$ and $^{236}U/^{238}U$ ratios for sample 8802D combined with the fact that the counterpart water sample unequivocally contains depleted U argue strongly that this sample also contains a small component of depleted U. Evidence that W23L08 filter contains anthropogenic U comes only from the $^{238}U/^{235}U$ ratio. Whereas we feel that this evidence is strong, we recommend additional analyses to confirm this conclusion.

The rest of the samples have natural $^{238}U/^{235}U$ ratios and $^{236}U/^{238}U$ ratios are considered below detection (see discussion below). $^{234}U/^{238}U$ ratios for the aquifer samples are typical for this part of the INEEL. There are no other equivalently analyzed $^{234}U/^{238}U$ ratios for vadose zone or perched water samples from the INEEL. With the exception of the samples discussed above, the $^{234}U/^{238}U$ ratios of vadose zone and perched water samples collected for this study are considered natural.

3.2 Plutonium

239-Plutonium is expected to be the most abundant isotope of Pu in this study, and therefore the most readily detectable. The highest ^{239}Pu abundance measured for all sample is approximately 5E7 atoms (equivalent to approximately 1.7 femtocurries of Pu with a $^{240}Pu/^{239}Pu$ ratio of 0.18 [in all subsequent conversions of atoms to curries a $^{240}Pu/^{239}Pu$ ratio of 0.18 is assumed]) per sample. Most samples have considerably lower ^{239}Pu abundances. All of these measurements

are near the detection limits of our procedures. Therefore, none of the samples yield unambiguous evidence for the presence of Pu. The results were therefore rigorously examined to determine the statistical significance of the data and to place reasonable confidence levels regarding the presence of Pu in these samples.

3.2.1 ^{239}Pu Concentration

Table 2 and Figure 5 present the results and plots of Pu data reduced as discussed above. In Figure 5a and 5b, the data are ranked in order of decreasing ^{239}Pu abundances. Figure 5a shows that two samples, TW1 filter and 8802D filter plot well above the rest of the samples even given their two-sigma uncertainties. Another four samples describe a break in the data, plotting above the line described by the rest of the data. However, the two-sigma uncertainties of these data overlap with two sigma uncertainties of the bulk of the data. A plot of ^{239}Pu atoms versus the inverse of the normal cumulative distribution (Fig. 5b) was constructed to examine if the data set is truly a non-random set of numbers, i.e. that these six data points form a distinct population. In such a plot, a normal Gaussian distribution will follow a straight line. The plot shows a pattern similar to that of Figure 5a with the same two samples (TW1 filter and 8802D filter) plotting well above the rest and the same four samples forming a break in the line formed by the remainder of the samples. It must be pointed out that, in both plots, a few of the samples plot *below* the trend shown by the bulk of the samples. This indicates that our methods have produced anomalously low values, further complicating the significance of the higher values.

Another test was performed to evaluate the statistical significance of the data. Given the calculated ^{239}Pu and the assigned uncertainty, the test evaluates the number of times a value that is as many or greater number of standard deviations from the mean would be expected to occur.

This test assumes that the measurements all have the same mean (in this case 0) and are normally distributed. Results of the test are presented in Table 2 and shown in Figure 5c. The test shows that the two samples with high Pu are not expected occurrences in this data set. Sample PA03 is expected to occur <0.2 times in this data set. The other samples with suspected high values of Pu (PA02, SDA8, and W23L08) are predicted to occur in this data set and thus are not regarded as anomalous by this statistical test. The test is very sensitive to uncertainties of the data. Therefore the order of the data changes slightly and, in addition, W23L08 moves down in rank whereas M1S filter and USGS119 filter move up in rank.

The above statistical evaluations yield consistent results that add confidence to the interpretation of the data set. All statistical tests show that samples TW1 filter and 8802D filter contain ^{239}Pu at levels that are statistically above background for the analytical technique. These two samples, as well as TW1 water also show unambiguous proof of anthropogenic U. The suggestion that Pu has migrated into the vadose zone is consistent with the evidence that U has migrated at these localities. Finally, the Pu detections for these samples are in the particulate phase, which is to be expected due to the low solubility of Pu and its tendency to adsorb onto particulate material (Langmuir, 1997). Therefore, although the Pu results are not unambiguous, several lines of evidence taken together strongly suggest the presence of Pu in these samples.

One additional sample, PA03 filter, consistently ranks highly in all statistical tests. Anthropogenic U was not detected in this sample. Although the data presented here do not prove the presence on ^{239}Pu in this sample, they do suggest the possibility that Pu is present.
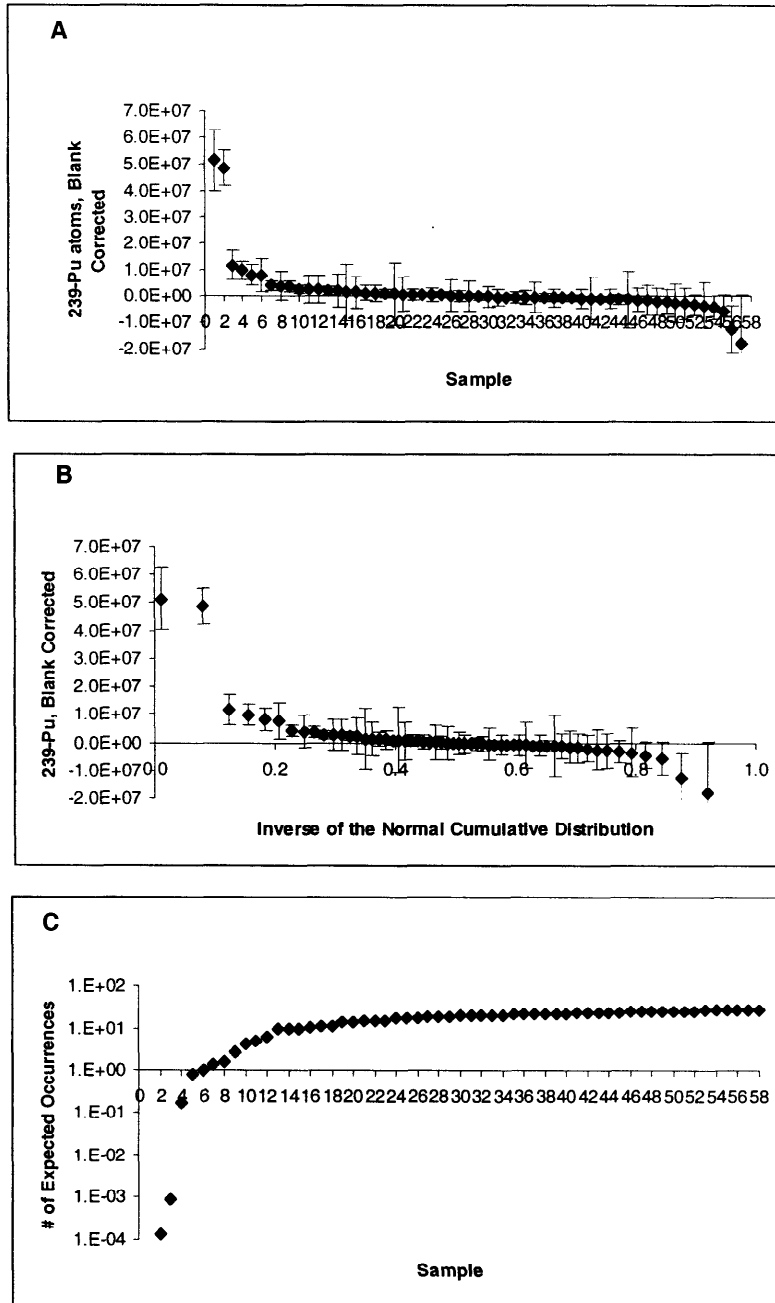
Figure 5. A and B show plots of ^{239}Pu abundance, ranked in decreasing order for all samples (A) and versus the Inverse of the Normal Cumulative Distribution (B). Two sigma uncertainties are shown. Sample rank is given in Table 2. In both plots, sample 58 plots below the limits of the y axis. Note that TW1 filter and 8802D filter (samples 1 and 2) plot far above the remainder of the samples and the break defined by samples 3-6. C is a plot of the number of expected occurrences for this sample set (see text for description). Sample 1 (8802D) plots well below the minimum value shown on the y axis. Sample number identifiers are keyed to Table 2.

Samples PA02 filter, SDA8 filter, W23L08 filter, M1S filter, and USGS119 filter rank highly in at least one statistical test. Of these, only W23L08 filter shows evidence for anthropogenic U. We conclude that the likelihood that these samples contain ^{239}Pu is very slight, but recommend that these samples be included in future surveys.

The data are interpreted to indicate that all other samples do not contain ^{239}Pu at detectable levels of about 5E6 atoms per sample (approximately 0.2 femtocurries per sample). For all aquifer samples and the river sample, >1.5 L of water was analyzed and the filter sample consists of material removed from 2.5-3.0 L of water. Thus, the stated upper limit for ^{239}Pu abundance is actually less when compared to a 1L sample size. Sample volumes for all vadose zone samples were lower, but for most the detection limit for ^{239}Pu is still less than 5E7 atoms/L of water (approximately 2 femtocurries/L of water).

Four of the samples are not included in the above analysis and plots. As discussed above, sample M7S was run on a different instrument, therefore the data were processed separately. No Pu was detected in this sample. Due to poor chemical yield, a detection limit of approximately 2.9E7 atoms is assigned to this analysis. Three additional samples (M1S water, M10S water, and USGS117 water) also had poor chemical yields so that the data were not processed with the remainder of the samples. These samples also showed no evidence of Pu with a detection limit of approximately 5E8 atoms/sample (approximately 15 femtocurries/sample).

3.2.2 ^{240}Pu/^{239}Pu Isotopic Ratios

The ^{240}Pu/^{239}Pu ratio can be useful to fingerprint the source of Pu and, in the case of this study, may possibly help to distinguish true occurrences of Pu from background. The ^{240}Pu/^{239}Pu for

most Pu varies between about 0.06 for weapons-grade Pu to about 1.0 for Pu produced in a reactor with high burn-up. Most reactor-produced Pu has a ^{240}Pu/^{239}Pu ratio of less than 0.5. Plutonium that was released into the atmosphere during nuclear testing has an average isotopic ratio of 0.18 (Shalkovitz, 1983). This is commonly referred to as "fallout Pu" and is found in most surficial soils globally. In this study, all of the ^{239}Pu measurements are near or below analytical background, therefore measurements of the smaller ^{240}Pu signal are even less precise and identifying true detections is even more problematic. Therefore only ^{240}Pu/^{239}Pu ratios for the two samples with the most likely ^{239}Pu detections are discussed below. The ^{240}Pu measured for the remainder of the samples is considered below detection.

Samples TW1 filter and 8802D filter have ^{240}Pu/^{239}Pu ratios of 0.17 ± 0.14 and 0.05 ± 0.06 respectively. The high uncertainty of the ^{240}Pu/^{239}Pu ratio for sample TW1 filter encompasses the range of weapons-grade Pu, fallout Pu and reactor-produced Pu. The ^{240}Pu/^{239}Pu for sample 8802D filter is better constrained (0.0 to 0.11) and implies at least a component of weapons-grade Pu. The fact that both ratios fall within the expected range of ^{240}Pu/^{239}Pu ratios helps support the contention that the ^{239}Pu detections are, in fact, real.

4.0 Discussion

4.1 ^{236}U/^{238}U Ratios

With the exception of sample TW1 water and filter and 8802D water, all the samples have very low measured ^{236}U/^{238}U ratios (all less than 2.7E-6). These ratios do not necessarily reflect actual abundance of ^{236}U relative to ^{238}U. The measured ^{236}U in these samples (between 0 and about 2 counts per second) may reflect numerous effects, for example transmission of isobaric interferences, which are difficult to accurately quantify. Negative values are due to excessive

background corrections, which are also likely caused by interferences. In some of the samples, the $^{236}U/^{238}U$ ratios are very imprecise indicating variable ^{236}U count rates that are likely due to variable within-run transmission of isobaric interferences. In other cases, the measured $^{236}U/^{238}U$ ratios were more stable as shown by lower relative uncertainties. Although such data could signal the presence of ^{236}U, they could also reflect stable transmission of interferences. For this report, $^{236}U/^{238}U$ ratios less than 3E-6, when taken alone, are considered below detection.

The presence of ^{236}U is unequivocally documented for samples TW1 water and filter and 8802D water. As discussed above, the $^{236}U/^{238}U$ ratio for sample 8802D filter may reflect the presence of very small amounts of ^{236}U. The fact that ^{236}U is found in samples that contain both enriched and depleted U is expected and reflects the fact that the U selected for further enrichment and/or depletion was previously modified within a nuclear reactor.

4.2 $^{234}U/^{238}U$ Ratios in Water Samples

234-Uranium ($t_{1/2}$=2.48 x 10^5 y) is part of the ^{238}U ($t_{1/2}$=4.47 x 10^9 y) radioactive decay series. The $^{234}U/^{238}U$ ratio in rocks is generally close to the secular equilibrium value of approximately 55 x 10^{-6} (55 ppm). However, $^{234}U/^{238}U$ ratios in groundwater is typically greater than the secular equilibrium value because of preferential dissolution of ^{234}U from crystallographic defects created by alpha recoil and because of direct ejection of ^{234}U into groundwater by recoil (see Gascoyne, 1992 for review and additional hypotheses). Natural variations in groundwater $^{234}U/^{238}U$ reflect the competing effects of decreased ^{234}U due to radioactive decay, addition of ^{234}U by selective leaching and recoil, and addition of U with equilibrium $^{234}U/^{238}U$ isotopic ratios by rock and/or mineral dissolution. $^{234}U/^{238}U$ ratios in INEEL groundwater range from 0.000086

to 0.000168 in the shallow aquifer beneath INEEL (Roback et al., in review), but much higher ratios have been observed in groundwater elsewhere (e.g. Osmond and Cowart, 1992). Most rock and especially rock greater than 1 my has ^{234}U/^{238}U ratios that are near secular equilibrium.

The high ^{234}U/^{238}U ratios measured for samples TW1 water and filter do not by themselves signal the presence of anthropogenic U. However, the ^{234}U/^{238}U ratios measured for theses samples are a factor of 3.5 times higher than have been thus far measured in a large number of INEEL samples. The ^{234}U/^{235}U ratios for these samples range from 0.0079 to 0.011, values that are well within the range of natural samples at INEEL. These data coupled with the ^{238}U/^{235}U and ^{236}U/^{238}U data indicate that these high ^{234}U/^{238}U ratios are not due to natural enrichment effects, but rather, are due to depletions in ^{238}U relative to ^{234}U. The measured ^{234}U/^{238}U for TW1 samples most likely reflect mixtures of enriched U and natural U. Mixing calculations using reasonable values for the natural end member suggest that the ^{234}U/^{235}U ratios of the enriched end member are not more than 20% from natural values. Thus, processes used to enrich U detected at TW1 did not greatly affect the relative abundances of ^{234}U and ^{235}U.

130

125 — BLR (Roback et al., in review)
North of SDA → ○
M3S
120 — ◆M7S
M14S ◆
USGS 87
115 — ○
BLR-99A

South boundary
of SDA

USGS 117 --- USGS 119
◆
M1S
M10S

234U/238U (ppm)

110 —
◆
USGS 120

105 —
◆
USGS 89 (Roback et al., in review)

100
0.0   0.2   0.4   0.6   0.8   1.0   1.2   1.4

1/[U] ppb
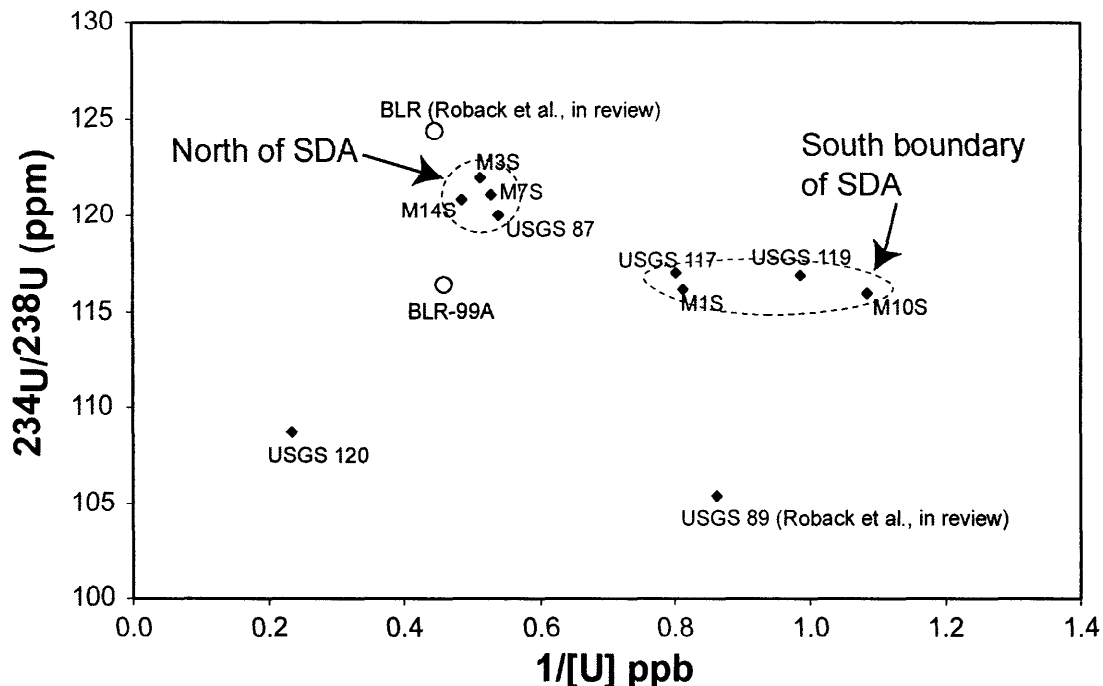
Figure 6. Plot of $^{234}U/^{238}U$ ratios versus the reciprocal of U concentration for aquifer samples near the SDA. Samples north and south of the SDA have distinct U chemistry. It is possible that samples to the north of the SDA were derived largely from the Big Lost River. Samples nearest the spreading areas (USGS 120 and USGS 89) have U chemistry that is distinct from the other samples.

U is readily soluble in oxidizing groundwater (Gascoyne, 1992 and references therein) and therefore it can be used as a relatively conservative tracer, particularly in rapidly flowing aquifers. Roback et al. (in review) used $^{234}U/^{238}U$ ratios to delineate regional flow patterns and mixing relations in the aquifer beneath the INEEL. Most $^{234}U/^{238}U$ ratios determined for this study are in good agreement with values reported in Roback et al. (in review) and help to support their conclusions regarding regional flow patterns.
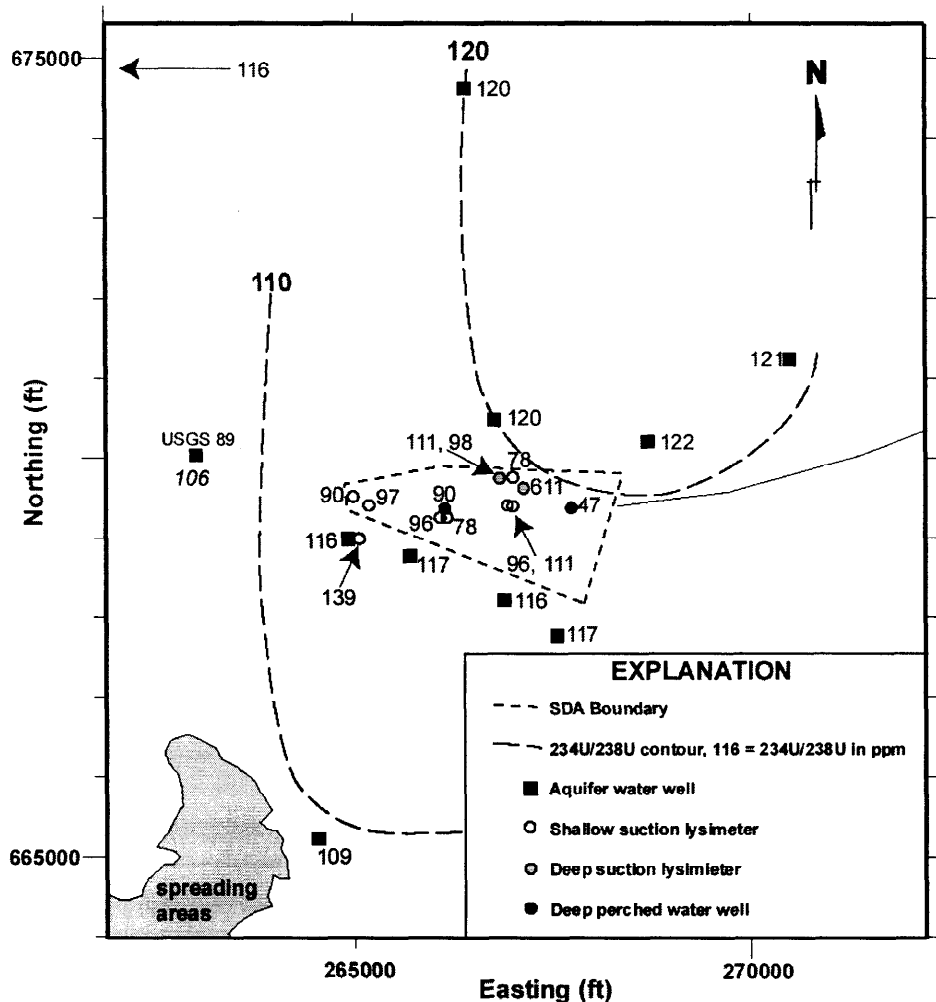
Figure 7 Map

675000 ◄———— 116

120
■ 120

N

110

121■

USGS 89
■
106

120
■ 120

111, 98
▲ 78
◯◯ ◯61
● 122

90◯◯97   90
96 ▲ 78
116■◯   ◯◯   ●47
■117
139   ■96, 111
■116

■117

EXPLANATION

– – – SDA Boundary

—— 234U/238U contour, 116 = 234U/238U in ppm

■ Aquifer water well

◯ Shallow suction lysimeter

◯ Deep suction lysimeter

● Deep perched water well

665000 ■ 109

spreading areas

265000   **270000**

Easting (ft)

Northing (ft)

Figure 7. Map showing $^{234}U/^{238}U$ isotopic ratios. Datum from USGS 89 is from Roback et al. (in review). The sample of Big Lost River (BLR-99A) was collected to the west of the arrow at easting approximately 262500 ft. See text for discussion.

U concentration data combined with the $^{234}U/^{238}U$ ratios measured for the aquifer water samples in this study show an interesting and potentially informative geographic pattern (Figs. 6 and 7). Samples to the north and east of the SDA have higher concentrations (1.86 to 2.06 ppb) than do samples just outside of the southern boundary of the SDA (U concentrations of 0.92 to 1.25 ppb). All aquifer samples in this study have a fairly narrow range of $^{234}U/^{238}U$ ratios between 109 ppm and 121 ppm and these ratios generally increase from the southwest to northeast. The four samples to the north and northeast of the SDA have internally consistent $^{234}U/^{238}U$ ratios and U

concentration. These data indicate that the water mass to the north and northeast of the SDA is quite homogeneous in terms of U chemistry, which may point to similar sources, thorough mixing, and/or similar geochemical evolution. The $^{234}U/^{238}U$ ratios and U concentration of the four samples to the north and northeast of the SDA differ from those from wells along the southern boundary of the SDA, which have lower isotopic ratios as well as U concentrations, indicating that this water mass is geochemically distinct from that to the north and east of the SDA, at least in terms of its U chemistry. The differences in U chemistry between these two sample groups may reflect differences in source and/or chemical evolution that is related to their proximity to the spreading areas and/or the Big Lost River. A test of these hypotheses awaits collection and evaluation of additional geochemical and isotopic data from this area.

4.3 Comparison of Water and Filter Isotopic Data

Comparison between water and filter isotopic data may shed light on the mode of transport of U and Pu, i.e. whether they are carried as dissolved constituents or as particles. All the samples were filtered at 0.5 µm, thus splitting the sample into two aliquots, 1) constituents greater than 0.5 µm (referred to as the particulate fraction) and 2) constituents less than 0.5 µm. The generally accepted maximum size of colloids is 0.45 µm, and they typically exist as much smaller particles. The methods employed for this study did not attempt to separate dissolved from colloidal constituents and, as a result, data presented herein cannot differentiate between whether U and Pu were carried as a dissolved or colloidal phase. In the following, the combined dissolved and colloidal phases will be simply referred to as the dissolved phase. Uranium isotopic data for the paired water and filter samples are shown in Figures 2, 3, and 4. As concluded above, all possible occurrences of Pu are from the particulate fraction.

Samples TW1 water and filter have analytically indistinguishable U isotopic data indicating that

isotopic equilibrium has been achieved between dissolved and particulate fractions. The filter

sample from 8802D has $^{238}U/^{235}U$ and $^{236}U/^{238}U$ isotopic ratios that are much closer to that of

natural U than data from the counterpart water sample. This likely indicates that the filter sample

is dominated by particles with natural U isotopic composition. This is to be expected given that

the water from this sample has anomalously low U concentration and therefore low capacity to

affect the U isotopics of the particles through adsorptive exchange. The only other sample with a

high probability of containing non-natural U is W23L08 filter, which has a $^{238}U/^{235}U$ ratio that is

slightly elevated from natural. In contrast, the corresponding water sample has natural U

isotopics suggesting that U is being transported in the particulate rather than the dissolved phase

at this locality.

In all but two of the samples, the $^{234}U/^{238}U$ ratios of the filter samples are either lower than, or

essentially equal to, the corresponding water samples. Lower $^{234}U/^{238}U$ isotopic ratios for the

filter samples likely indicates that the waters are carrying rock particles with $^{234}U/^{238}U$ close to

secular equilibrium, in addition to U that was adsorbed from the water. Samples for which the

$^{234}U/^{238}U$ ratios of the filter and water are nearly identical likely carry most of the particulate U as

an adsorbed and therefore readily exchangeable phase. Five of the ten aquifer samples, the river

sample, and one of the perched water samples have lower $^{234}U/^{238}U$ ratios for the filter when

compared to the water. In contrast, with only two exceptions, all vadose zone filter and water

samples have nearly identical $^{234}U/^{238}U$ ratios. This result is expected for a number of reasons:

1) the aquifer and river samples have a higher capacity to carry more and coarser rock fragment

than do the vadose zone samples due to greater flow velocities, 2) the vadose zone samples were

collected via suction lysimeters, which filter out some of the coarse particles, and 3) for USGS

92, the collection method entrained a large amount of sediment. Samples W25 and 8802D do

not follow this pattern, i.e. the water samples have lower $^{234}U/^{238}U$ than do the corresponding

filter samples. Sample W25 yielded the lowest mass of U on the filter of all the samples. The

uncertainties of the data for this sample are very high due to the large blank correction, and may

in fact be understated. The $^{234}U/^{238}U$ ratio of W25 filter should therefore be treated

conservatively. Sample 8802D water has depleted $^{238}U/^{235}U$ isotopics. A likely explanation for

the fact that the water sample has a lower $^{234}U/^{238}U$ ratio than the filter is that the $^{234}U/^{238}U$ ratio

is lowered due to the presence of enriched U.

All possible Pu detections are from the filter samples indicating that Pu was likely transported as

a particle rather than a dissolved constituent. This is to be expected owing to the generally low

solubility of Pu in groundwater and its affinity to adsorb onto particles (Langmuir, 1997). This

study does not provide evidence that Pu was transported as a colloid (e.g. Kirsting et al., 1999).

We cannot rule out the possibilities that true colloids coagulated between collection and

processing to form coarser particles, or that the colloids adsorbed onto the filter or filtered

particles during filtration.

5.0 Recommendations

This study documents that anthropogenic U and probably Pu are present in two sampling

localities: TW1 and 8802D. Continued monitoring of these will help to confirm the presence for

Pu as well as document changes in the anthropogenic contribution to the U in the sample. The

anomalously low U concentration for sample 8802D may suggest reducing conditions brought on

by contaminant release. Additional geochemical evaluation of this well should be conducted to investigate redox conditions, and the possible role of organic complexing agents in contaminant transport. Two other samples, W23L08 and PA3, yielded probable or possible detections for anthropogenic U and Pu respectively. Additional analyses of samples from these localities are necessary to positively document the presence of anthropogenic U or Pu. In addition to replicate analyses, processing of larger samples would aid in confirming Pu detections in these four samples and possibly in documenting the presence of Pu in other samples.

Natural ^{234}U/^{238}U ratios in aquifer groundwater are useful indicators of flow and geochemical evolution. These data give us clues to physical and chemical interactions of the groundwater sources, water rock interactions and mixing relations (e.g. Roback et al., in review). The pattern of U isotopic ratios and concentrations determined for this study reveals distinct special patterns in the vicinity of the SDA. Such data from a more complete coverage of aquifer wells coupled with other data, such as major element concentration data and other isotopic data, would add greatly to our understanding of local groundwater sources, flow patterns, and geochemical evolution in the vicinity of the SDA. Ultimately these data would lead to better prediction of the migration of contaminants from the SDA.

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